

PATENT ABSTRACTS OF JAPAN

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(54) COMPOSITION FOR POLYURETHANE**(57)Abstract:**

PURPOSE: To provide the title composition which is lowly viscous and easily handleable and can give a cured polyurethane product excellent in water resistance and mechanical strengths.

CONSTITUTION: The composition is prepared by mixing a polyester polyol prepared by esterifying a mixed diol comprising 70-45wt.% 1,9-nonanediol and 30-55wt.% 2-methyl-1,8-octanediol with a dicarboxylic acid component based on hydrogenated dimer, a diisocyanate compound and a chain extender.

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CLAIMS

[Claim(s)]

[Claim 1] The constituent for polyurethane with which the polyester polyol, the diisocyanate compound, and chain extension agent which make it come to esterify the mixed diol which consists of 70 - 45 % of the weight of 1 and 9-nonane diols and 30 - 55 % of the weight of the 2-methyl -1 and 8-octanediol with the dicarboxylic acid which makes a hydrogenation dimer acid a principal component were blended.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] this invention relates to the constituent for polyurethane which gives the polyurethane hardened material which is excellent in handling workability with hypoviscosity, and is excellent in a mechanical strength and durability.

[0002]

[Description of the Prior Art] As one raw material of a polyurethane resin, generally the polyester polyol is used and the general-purpose polyester polyol is an adipic-acid system polyester polyol. However, this adipic-acid system polyester polyol had the problem that it was inferior to durability, although a mechanical property and oil resistance were good. Although the polyester polyol which used dodecane diacid as a dicarboxylic acid for waterproof enhancement is proposed, since dodecane diacid is expensive, generally it has come to be used.

[0003] On the other hand, it replaces with dodecane diacid, there is also a proposal of the polyester polyol using the hydrogenation dimer acid, and the polyester polyol compounded from diols, such as a hydrogenation dimer acid and 1 and 4-cyclohexane dimethanol, is proposed as a polyester polyol which gives polyurethane excellent in durability (JP,4-145118,A).

[0004] However, since the polyester polyol which the polyester polyol compounded from alicyclic diols, such as a hydrogenation dimer acid and 1 and 4-cyclohexane dimethanol, has the problem that it is inferior to workability as compared with the polyester polyol which used the aliphatic diol since hyperviscosity, and consists of an aliphatic diol and a hydrogenation dimer acid on the other hand had inadequate alkali resistance, for the intended use of coating etc., its fitness was not quite satisfactory.

[0005]

[Means for Solving the Problem] In order that this invention persons may solve the above-mentioned technical problem, as a result of inquiring zealously, the dicarboxylic acid which makes a hydrogenation dimer acid a principal component as a dicarboxylic acid. Moreover, the mixed diol which consists of 70 - 45 % of the weight of 1 and 9-nonane diols and 30 - 55 % of the weight of the 2-methyl -1 and 8-octanediol as a diol is used, respectively. The polyurethane hardened material with which the polyester polyol which is made to carry out the polycondensation of these and is obtained is hypoviscosity, and is moreover obtained using it finds out excelling in durability and a mechanical strength extremely, and came to complete this invention.

[0006] That is, this invention is the constituent for polyurethane with which the polyester polyol, the diisocyanate compound, and chain extension agent which make it come to esterify the mixed diol which consists of 70 - 45 % of the weight of 1 and 9-nonane diols and 30 - 55 % of the weight of the 2-methyl -1 and 8-octanediol with the dicarboxylic acid which makes a hydrogenation dimer acid a principal component were blended. Hereafter, it explains still in detail about this invention.

[0007] An oleic acid and linolic acid are mentioned as a typical unsaturation aliphatic carboxylic acid which the hydrogenation dimer acid in this invention hydrogenates the dimer acid whose carbon number is the dimer of the unsaturation aliphatic carboxylic acid of 18, and is obtained and which is a high-class aliphatic dicarboxylic acid and serves as the raw material of the above-mentioned dimer acid. Since the dimer acid compounded from an oleic acid or linolic acid is marketed from Henkel Hakusui, Inc. and U.S. Emily, it can obtain a hydrogenation dimer acid by hydrogenating the dimer acid of these marketing under the hyperbaric pressure. Moreover, you may use the hydrogenation dimer acid of marketing by the ***** international company, such as tradename PRIPOL-1009 or PRIPOL-1008. A desirable hydrogenation dimer acid is the thing of acid-number;194-198, less than [iodine number;10], and saponification value;196-200.

[0008] In this invention, other dicarboxylic acids can be used with the above-mentioned hydrogenation dimer acid, and aromatic dicarboxylic acids, such as aliphatic dicarboxylic acids, such as an adipic acid, an azelaic acid, a sebacic acid, and dodecane diacid, or a terephthalic acid, and an isophthalic acid, are mentioned by request as such a dicarboxylic acid. The amount of dicarboxylic acids used other than a hydrogenation dimer acid is the waterproof point of the polyurethane obtained, and less than [of the whole quantity of a dicarboxylic-acid component / 10 mol %] is desirable. In order to obtain the polyurethane which is highly excellent in durability, it is desirable that the whole quantity of an acid component is a hydrogenation dimer acid.

[0009] The polyester polyol in this invention is polyester which is made to carry out the polycondensation of the dicarboxylic acid which makes the above-mentioned hydrogenation dimer acid a principal component to the mixed diol which consists of 70 - 45 % of the weight (henceforth ND) of 1 and 9-nonane diols, and 30 - 55 % of the weight of the 2-methyl -1 and 8-octanediol (henceforth MOD), and is obtained and which has a hydroxyl in the both ends of a molecule. In the mixed diol by which the rate of ND exceeds 70 % of the weight, the polyester diol obtained is a solid-state-like, and the polyurethane which is inferior to workability and is obtained from this polyester diol is inferior to tensile strength. On the other hand, when the rate of ND is less than 45 % of the weight, the elongation percentage of the polyurethane obtained is too small. A more desirable mixed diol is

mixture (ND;65-50 % of the weight and MOD;35-50 % of the weight).

[0010] After the polycondensation of the carboxylic acid which makes the above-mentioned hydrogenation dimer acid a principal component, and a mixed diol carries out the temperature up of the raw material mixture even to 220-250 degrees C in inert gas, removing the same technique as the case where common polyester is manufactured, i.e., the water to generate, out of the system of reaction, it can be performed by the technique of removing water and the diol taught superfluously under reduced pressure. In this invention, the desirable average molecular weight of a polyester diol is the number average molecular weight of the polystyrene conversion by the gel permeation chromatography, and is 1,000-2,000.

[0011] The constituent for polyurethane of this invention is a constituent with which compounds, such as polyhydric alcohol as the diisocyanate compound and chain extension agent as a curing agent or a multiple-valued amine, were blended with the above-mentioned polyester diol, and the following compounds are mentioned as a diisocyanate compound. For example, it is aliphatic series or alicyclic group diisocyanate, such as aromatic diisocyanate, such as diphenylmethane diisocyanate, 2, 4-tolylene diisocyanate, 2, 6-tolylene diisocyanate, phenylene diisocyanate, 1, and 5-naphthalene diisocyanate and xylylene diisocyanate, and hexamethylene G ******, isophorone diisocyanate, 4, and 4'-dicyclohexylmethane diisocyanate, hydrogenation-ized xylylene diisocyanate, and tetramethyl xylenediisocyanate, etc. Preferably, they are diphenylmethane diisocyanate, hexamethylene G ******, and isophorone diisocyanate.

[0012] As polyhydric alcohol for chain extension agents, ethylene glycol, a propylene glycol, 1, 4-butanediol, neopentyl glycol, 1, 4-cyclohexane dimethanol, 1, 6-hexandiol, 1, 9-nonane diol, the 2-methyl -1, 8-octanediol, 1, 12-dodecane diol, a dimer diol, a trimethylol propane, a glycerol, etc. are mentioned. as a multiple-valued amine Ethylenediamine, a propylenediamine, a diethylenetriamine, a piperazine, a phenylenediamine, an isoholondiamine, a tolylenediamine, etc. are mentioned.

[0013] As for each component, in the constituent for polyurethane of this invention, it is desirable to use per one mol of aforementioned polyester polyols and a chain extension agent at a rate of 1-4 mols, and, as for the amount of the diisocyanate compound used, it is desirable that it is a grade mol mostly to the total quantity of a polyester polyol and a chain extension agent.

[0014] Although its technique of adding the aliphatic diol of a chain extension agent is desirable after it warms it at about 50-100 degrees C after the manufacture of the constituent for polyurethane mixed the whole quantity of a polyester polyol and a diisocyanate compound, and it combines an isocyanate machine with the terminal of a polyester polyol, it is good also by the technique of mixing simultaneously a polyester polyol, diisocyanate, and a chain extension agent.

[0015] In case of a manufacture of the above-mentioned constituent, the organic solvent may be used together and a methyl ethyl ketone, ethyl acetate, toluene, a dioxane, a dimethylformamide, dimethyl sulfoxide, etc. are mentioned as a desirable solvent. When using the constituent for polyurethane of this invention for intended use, such as a polyurethane foam, it is desirable to use the constituent which does not contain a solvent.

[0016] It is 80-150 degrees C to perform the hardening reaction of the constituent for polyurethane of this invention at ordinary temperature -200 degree C desirable still preferably. In order to promote a reaction in case of hardening, you may use tin system catalysts, such as amine system catalysts, such as a triethylamine, N-ethyl morpholine, and a triethylenediamine, or trimethyl tin laurate, and dibutyltin dilaurate. Moreover, the various stabilizers for a coloring agent and weatherability, and heat-resistant enhancement may be added to the constituent for polyurethane as occasion demands, and this constituent is used for it suitable for intended use, such as coating, adhesives, artificial leather, a synthetic leather, fiber, a form, and rubber.

[0017]

[Example] Hereafter, by showing the example of reference, an example, and the example of a comparison explains this invention still concretely.

To the reactor of <example 1 of reference> (manufacture of polyester polyol A) 1L the product made from hydrogenation dimer-acid [***** International -- PRIPOL-1009 -- 290g And the rate of 1 and 9-nonane diol, and the 2-methyl -1 and 8-octanediol teaches mixed diol 400g of 65:35 (weight %), after applying to 240 degrees C for 1 hour and carrying out a temperature up to them from a room temperature under the nitrogen ambient atmosphere, it was made to react at 240 degrees C for 6 hours, and the water generated in the meantime was made to ****. Subsequently, the polyester polyol which has the property of a publication in Table 1 was obtained by making a superfluous diol **** for 30 minutes under reduced pressure of 2mmHg.

[0018] As a <example 2 of reference> (manufacture of polyester polyol B) mixture diol, except that the rate of 1 and 9-nonane diol, and the 2-methyl -1 and 8-octanediol used the mixed diol of 50:50 (weight %), the polyester polyol was obtained like the example 1 of reference.

[0019] The polyester polyol was compounded by the same technique as the example 1 of reference using hydrogenation dimer-acid [which was used in the example 1 of the <examples 3-5 of reference> reference], 1, 4-cyclohexane dimethanol (henceforth CHDM), 1, and 9-nonane diol or 1, and 6-hexandiol (henceforth HD).

[0020]

[Table 1]

| 参考例 | 合成ポリエステル | 使用ジオール | 酸価 mgKOH/g | 水酸基価 mgKOH/g | 数平均 分子量 | 粘度 cP/25℃ |
|-----|----------|-------------------|---------------|-----------------|------------|--------------|
| 1 | A | ND/MOD (65/35) | 0.9 | 103 | 1090 | 2,300 |
| 2 | B | ND/MOD (50/50) | 0.9 | 105 | 1070 | 2,400 |
| 3 | C | CHDM | 1.0 | 75 | 1500 | 34,500 |
| 4 | D | ND | 1.0 | 99 | 1130 | 2,400 |
| 5 | E | HD | 0.7 | 93 | 1210 | 2,200 |

[0021] A; 150g (0.14 mols) of the polyester polyols obtained in the example 1 of <example 1> reference and diphenylmethane diisocyanate; 103.4g (0.41 mols) were taught to the 500ml flask, and it was made to react at 80 degrees C under the nitrogen ambient atmosphere for 2 hours. subsequently -- a chain extension agent ***** -- 1 -- [4-butanediol; 24.8g (0.28 mols)], it stirred for 30 seconds and the uniform transparent constituent for polyurethane was obtained The polyurethane hardened material was obtained by heating the obtained constituent at 100 degrees C for 24 hours. By covering this hardened material over a heat press at the temperature of 180 degrees C, the dumbbell specimen was created and the tensile strength and the elongation at the time of fracture were measured (based on JISK7113). For the purpose of a waterproof examination, the above-mentioned dumbbell was measured also about tensile strength and elongation after being under a boiling water for one week. A result is as Table 2.

[0022] The polyurethane hardened material was obtained like the example 1 using the polyester polyol which consists of a diol of this invention obtained in the examples 3-5 of the <examples 1-3 of comparison> reference out of range. In the example 1 of a comparison which used polyester polyol C, since only the hardened material whose interior is not hardened was obtained even if it had hardened, about the hardened material of this example, measurement of physical properties of a front face was not completed. The result of other examples is as given in Table 2.

[0023]

[Table 2]

| | ポリエステル | 初期物性 | | 沸水浸漬後 | |
|-------|--------|--------|------|--------|------|
| | | 引張強度 | 破断伸び | 引張強度 | 破断伸び |
| 実施例 1 | A | 400 | 360 | 320 | 410 |
| 比較例 1 | C | —測定不可— | | —測定不可— | |
| 比較例 2 | D | 340 | 350 | 238 | 371 |
| 比較例 3 | E | 160 | 160 | 120 | 120 |

(単位 引張強度 ; Kg/cm² 破断伸び ; %)

[0024] Using <example 2> polyester polyol B, except having used hexamethylene di-isocyanate as diisocyanate, the polyurethane hardened material was obtained like the example 1, and the physical properties were measured. A result is as Table 3.

[0025] <Examples 4-5 of comparison> Polyester polyol C or polyester polyol E was used, and the polyurethane hardened material was obtained by the same technique as an example 2.

[0026]

[Table 3]

| | ポリエステル | 初期物性 | | 沸水浸漬後 | |
|-------|--------|-------|-------|-------|-------|
| | | 引張強度 | 破断伸び | 引張強度 | 破断伸び |
| 実施例 2 | B | 3 9 0 | 5 3 0 | 2 0 3 | 2 6 0 |
| 比較例 4 | C | 2 8 0 | 3 9 0 | 1 4 8 | 2 3 4 |
| 比較例 5 | E | 2 3 0 | 3 9 0 | 1 2 1 | 1 1 3 |

(単位 引張強度 ; Kg/cm² 破断伸び ; %)

[0027]

[Effect of the Invention] The constituent for polyurethane of this invention excels [viscosity] in handling workability low, and can also deal with a non-solvent. And according to this constituent, the polyurethane hardened material which is excellent in a mechanical strength and durability is obtained.

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(54) 【発明の名称】 ポリウレタン用組成物

(57) 【要約】

【目的】 低粘度で取り扱い作業性に優れ、かつ耐水性および機械的強度に優れるポリウレタン硬化物を与えるポリウレタン用組成物の提供。

【構成】 1, 9-ノナンジオール70~45重量%と2-メチル-1, 8-オクタンジオール30~55重量%からなる混合ジオールを水添タイマー酸を主成分とするジカルボン酸とエステル化させてなるポリエステルポリオール、ジイソシアネート化合物および鎖伸長剤が配合されたポリウレタン用組成物。

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【特許請求の範囲】

【請求項1】 1, 9-ノナンジオール70～45重量%と2-メチル-1, 8-オクタジオール30～55重量%からなる混合ジオールを水添ダイマー酸を主成分とするジカルボン酸とエステル化させてなるポリエステルポリオール-インシシアネート化合物および鎖伸長剤が配合されたポリウレタン用組成物。

【発明の詳細な説明】

[() ()]

【産業上の利用分野】本発明は、低粘度で取り扱い作業性に優れ、かつ機械的強度および耐水性に優れるポリウレタン硬化物を与えるポリウレタン用組成物に関するものである。

[0002]

【従来の技術およびその問題点】ポリウレタン樹脂の一原料として、ポリエステルポリオールが一般的に使用されており、最も汎用のポリエステルポリオールはアジピン酸系ポリエステルポリオールである。しかしながら、該アジピン酸系ポリエステルポリオールは、機械的性質や耐油性は良好であるが、耐水性に劣るという問題があった。耐水性の改良のために、シカルボン酸としてドデカン二酸を用いたポリエステルポリオールが提案されているが、ドデカン二酸が高価なために一般的に使用されるには至っていない。

【0003】他方、トデカン二酸に代えて、水添ダイマー酸を用いたポリエステルポリオールの特案もあり、耐水性に優れたポリウレタンを与えるポリエステルポリオールとして、水添ダイマー酸と1, 4-シクロヘキサンジメタノール等のシオールから合成されるポリエステルポリオールが提案されている(特開平4-145118号公報)。

【0004】しかしながら、水添ダイマー酸と1,4-シクロヘキサジエンメタノール等の脂環式シオールから合成されたポリエステルポリオールは、脂肪族シオールを用いたポリエステルポリオールと比較して高粘度なため、作業性におけるとい問題があり、一方脂肪族シオールと水添ダイマー酸からなるポリエステルポリオールは耐アルカリ性が不十分であるため、塗料等の用途には適性が一歩であった。

[0005]

【課題を解決するための手段】本発明者らは、上記課題を解決するために鋭意検討した結果、シカルボン酸として水添マイマー酸を主成分とするシカルボン酸を、またシオールとして、1，9-ノナンシオール70～45重量%と2-メチル-1，8-オクタジエノール30～55重量%からなる混合シオールをそれぞれ用い、これらを重縮合させて得られるポリエステルポリオールが、低粘度でありしかもそれを用いて得られるポリウレタン硬化物が、耐水性および機械的強度に極めて優れることを見出し、本発明を完成するに至った。

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【０００６】すなわち、本発明は、１、９－ジナジオール７０～４５重量％と２－メチル－１，８－オクタジオール２０～５５重量％からなる混合ジオールを水添ダイファー酸を主成分とするジカルボン酸とエステル化させてなるオリエステルポリオール、シイソシアネート化合物および鎖伸長剤が配合されたオリウレタン用組成物である。以下、本発明についてさらに詳しく説明する。

【0007】本発明における水添ダイマー酸は、炭素数が18の不飽和脂肪族カルボン酸の二量体であるダイマー酸を水素添加して得られる高級脂肪族カルボン酸であり、上記ダイマー酸の原料となる代表的な不飽和脂肪族カルボン酸としては、オレイン酸およびリノール酸が挙げられる。オレイン酸またはリノール酸等から合成されるダイマー酸は、ペンケル日本（株）や米国のエミリー社等からも市販されているので、これらの市販のダイマー酸を高圧下で水素添加することにより、水添ダイマー酸を得ることかできる。また、ユニファ・インターナショナル社製の商品名PRIPOL-1009またはPRIPOL-1008等の市販の水添ダイマー酸を使用しても良い。好ましい水添ダイマー酸は、酸価：194～198、ヨウ素価：10以下、ケン化価：196～200のものである。

【０００８】本発明においては、所望により、上記水添ダイマー酸と共にその他のシカルボン酸を使用でき、かかるシカルボン酸としては、アジピン酸、アセライン酸、セバシン酸およびトケラン二酸等の脂肪族シカルボン酸、またはテレフタル酸、イソフタル酸等の芳香族シカルボン酸が挙げられる。水添ダイマー酸以外のシカルボン酸の使用量は、得られるポリウレタンの耐水性の点で、シカルボン酸成分の全量の１０モル％以下が好ましい。高度に耐水性に優れたポリウレタンを得るためには、酸成分の全量か水添ダイマー酸であることが望ましい。

【０００９】本発明におけるポリエステルポリオールは、上記水添ダイマー酸を主成分とするシカルボン酸を、１，９－ノナンジオール（以下NDという）７０～４５重量％と２－メチル－１，８－オクタジオール（以下MODという）３０～５５重量％からなる混合ジオールと縮合させて得られる、分子の両末端にヒドロキシル基を有するポリエステルである。NDの割合が７０重量％を超える混合ジオールでは、得られるポリエステルンオールが固体状であって、作業性に劣り、かつ該ポリエステルンオールから得られるポリウレタンは引張強度に劣る。一方、NDの割合が４５重量％未満であると、得られるポリウレタンの伸び率が小さ過ぎる。より好ましい混合ジオールは、ND、６５～５０重量％およびMOD、３５～５０重量％の混合物である。

【0010】上記水添ダイマー酸を主成分とするシカルボン酸と混合シオールの重縮合は、一般的なポリエステルを製造する場合と同様な方法、すなわち、生成する水

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| 参考例 | 合成ポリエステル | 使用ジオール | 酸価 mgKOH/g | 水酸基価 mgKOH/g | 数平均 分子量 | 粘度 cP/25℃ |
|-----|----------|-------------------|---------------|-----------------|------------|--------------|
| 1 | A | ND/MOD (65/35) | 0.9 | 103 | 1090 | 2,300 |
| 2 | B | ND/MOD (50/50) | 0.9 | 105 | 1070 | 2,400 |
| 3 | C | CHDM | 1.0 | 75 | 1500 | 34,500 |
| 4 | D | ND | 1.0 | 99 | 1130 | 2,400 |
| 5 | E | HD | 0.7 | 93 | 1210 | 2,200 |

【0021】＜実施例1＞参考例1で得たポリエステルポリオールA：150g（0.14モル）、ジフェニルメタンジイソシアネート：103.4g（0.41モル）を500mlのフラスコに仕込み、窒素雰囲気下で、80℃で2時間反応させた。次いで、鎖伸張剤として、1,4-ブタンジオール：24.8g（0.28モル）を加え、30秒間攪拌して、均一な透明のポリウレタン用組成物を得た。得られた組成物を100℃で24時間加熱することにより、ポリウレタン硬化物を得た。該硬化物を温度180℃で熱プレスにかけることにより、ダンベル試験片を作成し、その引張強度および破断時の伸びを測定した（JISK7113に準拠）。耐水性試験の目的で、上記ダンベルを沸騰水に1週間浸漬した後※30

*の引張強度および伸びについても測定した。結果は表2のとおりである。

20 【0022】＜比較例1～3＞参考例3～5で得られた本発明の範囲外のジオールからなるポリエステルポリオールを用い、実施例1と同様にしてポリウレタン硬化物を得た。ポリエステルポリオールCを使用した比較例1では、表面は硬化していても内部が未硬化の硬化物しか得られなかったため、同例の硬化物については物性の測定ができなかった。その他の例の結果は、表2に記載のとおりである。

【0023】

【表2】

| | ポリエステル | 初期物性 | | 沸水浸漬後 | |
|------|--------|----------|------|----------|------|
| | | 引張強度 | 破断伸び | 引張強度 | 破断伸び |
| 実施例1 | A | 400 | 360 | 320 | 410 |
| 比較例1 | C | ——測定不可—— | | ——測定不可—— | |
| 比較例2 | D | 340 | 350 | 238 | 371 |
| 比較例3 | E | 160 | 160 | 120 | 120 |

(単位 引張強度：Kg/cm² 破断伸び：%)

【0024】＜実施例2＞ポリエステルポリオールBを用い、またジイソシアネートとしてヘキサメチレンジイソシアネートを用いた以外は、実施例1と同様にしてポリウレタン硬化物を得て、その物性を測定した。結果は表3のとおりである。

【0025】＜比較例4～5＞ポリエステルポリオールCまたはポリエステルポリオールEを使用して、実施例2と同様な方法により、ポリウレタン硬化物を得た。

【0026】

【表3】

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| | ポリエ テル | 初期物性 | | 沸水浸漬後 | |
|------|-----------|------|------|-------|------|
| | | 引張強度 | 破断伸び | 引張強度 | 破断伸び |
| 実施例2 | B | 390 | 630 | 203 | 260 |
| 比較例4 | C | 280 | 390 | 148 | 234 |
| 比較例5 | E | 230 | 390 | 121 | 113 |

(単位 引張強度: kgf/cm^2 破断伸び: %)

【0027】

【発明の効果】本発明のポリウレタン用組成物は、粘度が低く取扱作業性に優れており、無溶剤でも取り扱うこ

とができる。しかも、該組成物によれば、機械的強度および耐水性に優れるポリウレタン硬化物が得られる。